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(FILE 'HOME' ENTERED AT 12:38:41 ON 16 MAR 2009)
FILE 'CA' ENTERED AT 12:38:51 ON 16 MAR 2009
L1 24735 S PROTEIN(4A)(REMOV? OR FILTRAT? OR ULTRAFILTRAT? OR MICROFILTRAT? OR ULTRAMICROFILTRAT?) OR DEPROTEINAT?L2 10476 S L1 AND(FILTRAT? OR ULTRAFILTRAT? OR MICROFILTRAT? OR ULTRAMICROFILTRAT? OR FILTER? OR ULTRAFILTER? OR MICROFILTER? OR ULTRAMICROFILTER?)
L3 54 S L2 AND (ACETONITRILE OR CH3CN)
L4 30 S L3 AND PY<2003
L5 11 S L3 NOT L4 AND PATENT/DT
L6 41 S L4-5

=> d bib,ab l6 1-41

L6 ANSWER 14 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 137:43736 CA
TI The comparison of plasma deproteinization methods for the detection of low-molecular-weight metabolites by 1H nuclear magnetic resonance spectroscopy
AU Daykin, Clare A.; Foxall, Peta J. D.; Connor, Susan C.; Lindon, John C.; Nicholson, Jeremy K.
CS Biological Chemistry, Biomedical Sciences Division, Faculty of Medicine, Imperial College of Science, Technology and Medicine, London, SW7 2AZ, UK
SO Analytical Biochemistry (**2002**), 304(2), 220-230
AB Blood plasma is the major vehicle by which metabolites are transported around the body in mammalian species, and chem. anal. of plasma can provide a wealth of information relating to the biochem. status of an individual and is important for diagnostic purposes. However, plasma is very complex in physicochem. terms because it is composed of a range of org. and inorg. constituents with a wide range of mol. wts. and chem. classes and this makes anal. non-trivial. It is now well established that high-resoln. 1H NMR spectroscopy of blood plasma provides useful qual. and quant. biochem. information relating to metabolic disorders. However, one of the problems encountered in NMR spectroscopic anal. of blood plasma is the extensive peak overlap or presence of broad macromol. peaks in the 1H NMR spectrum, which can severely limit the amt. of obtainable information. Even with spectroscopic editing, information relating to low-mol.-wt. (MW) metabolites is frequently lost. Therefore, the efficiency of a range of conventional **protein removal** methods, in combination with the use of one- and two-dimensional NMR spectroscopic methods for evaluation, have been compared for the extn. of NMR-observable low-MW metabolites. It has been shown that these "deproteinization" methods vary considerably in recovery of low MW metabolites and a judicious choice is crucial for optimal extn. of a given analyte. The results presented here show that while **ultrafiltration** provides the "safest" method of plasma deproteinization, the signal-to-noise ratio of the resultant 1H NMR spectra is poor. On the other hand, **acetonitrile** pptn. at physiol. pH allows the detection of more low-MW metabolites and at higher concns. than any other method and provides the further advantages of being a rapid and simple procedure.

L6 ANSWER 17 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 132:87674 CA
TI Automated **protein** precipitation by **filtration** in the 96-well format
AU Biddlecombe, R. A.; Pleasance, S.
CS Department of International Bioanalysis, Division of Bioanalysis and Drug Metabolism, GlaxoWellcome R&D, Ware, UK
SO Journal of Chromatography, B: Biomedical Sciences and Applications (**1999**), 734(2), 257-265
AB The use of automated **protein** pptn. by **filtration** in the 96-well format as a rapid sample prepn. technique for high throughput bioanal. using liq. chromatog. tandem mass spectrometry is reported. A robotic sample processor is used to aspirate sequentially a plasma sample and **acetonitrile** sepd. by air gaps. These are then mixed by being dispensed into individual channels of a 96-well **filter** block. The resulting supernatant is sepd. from the pptd. plasma proteins by the application of gentle vacuum using a custom manifold. The **filtered** supernatants are collected into a deep well microtiter plate, evapd. to dryness using a heated 96-well dry down station and reconstituted in water prior to anal. The efficiency of the extn. procedure is measured by the Lowry method for detg. protein concn. This method was used to optimize both the vol. and the order of reagent addn., and to compare several prototype 96-well **filter** blocks. Using the optimized procedure a specific, precise and accurate method was developed for the β-agonist salbutamol in rabbit plasma with a calibration range of 1 to 100 ng/mL from 100 μl of sample.

L6 ANSWER 20 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 130:90010 CA
TI Therapeutic drug monitoring of doxorubicin in pediatric oncology using capillary electrophoresis
AU Hempel, Georg; Schulze-Westhoff, Petra; Flege, Silke; Laubrock, Nora; Boos, Joachim
CS Kinderklinik, Abteilung Haematologie/Onkologie, Univ. Muenster, Muenster, D-48129, Germany
SO Electrophoresis (**1998**), 19(16-17), 2939-2943
AB A method for the detn. of doxorubicin and its main metabolite doxorubicinol in blood plasma is described. 2 Different sample prepn. procedures are applied depending on the expected concn. To monitor the peak plasma levels, 10 μL of plasma are **deproteinated** with **acetonitrile**. After centrifugation, the supernatant is directly applied to the capillary by hydrodynamic injection. For the detn. of lower amts. of doxorubicin and doxorubicinol 100 μL of plasma is extd. by liq.-/liq. extn. with chloroform. After evapn. of the org. phase, the sample is reconstituted in **acetonitrile**/water (95/5 vol./vol.) and injected into the capillary by electrokinetic injection. Idarubicin serves as the internal std. Laser-induced fluorescence detection with an Ar-ion laser emitting at 488 nm and a 520 nm cut-off **filter** is used for detection. The accuracy of the method was calcd. to be 3.0% at higher concns. and 15.0% at the limit of quantification. Reproducibility data are in accordance to the generally accepted criteria for bioanal. methods. The

limit of quantification is 2 μg/L, enabling us to monitor doxorubicin plasma levels for several days after application. Noninvasive blood sampling (from the fingertip) using heparinized capillaries was a simple and convenient procedure and provides reproducible data. Initial results show high interindividual variability in doxorubicin peak plasma levels.

L6 ANSWER 22 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 126:248477 CA
OREF 126:47983a,47986a
TI A study of deproteinization methods for subsequent serum analysis with capillary electrophoresis
AU Ralston, Pamela B.; Strein, Timothy G.
CS Dep. Chem., Bucknell Univ., Lewisburg, PA, 17837, USA
SO Microchemical Journal (**1997**), 55(2), 270-283
AB Methods of serum **protein removal** are examd. for applicability with subsequent serum anal. for low mol. mass metabolites and pharmaceutical agents by capillary electrophoresis (CE). Each of the deproteinization methods considered here has been reported in the literature and successfully used for serum **protein removal** in other applications. Two of the methods are found to be superior to the others for CE anal. **Protein removal** by **ultrafiltration** with Centrifree Micropartition System **filters** and by pptn. using **acetonitrile** worked well for **protein removal**, as evidenced by reproducible (RSD < 6%) detns. of several low mol. wt. components. The former is capable of analyzing for free metabolites only, while the latter was found to be somewhat problematic with respect to evapn. of the **acetonitrile**, causing potential problems with reproducibility. Both techniques for **protein removal** were clearly suitable for the subsequent serum anal. by CE in untreated fused silica capillaries.

L6 ANSWER 24 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 125:8898 CA
OREF 125:2023a,2026a
TI Determination of pantothenic acid in infant milk formulas by high performance liquid chromatography
AU Romera, J. M.; Ramirez, M.; Gil, A.
CS Res. Dev. Dep., Abbott Laboratories S.A., Granada, 18004, Spain
SO Journal of Dairy Science (**1996**), 79(4), 523-526
AB A reverse-phase liq. chromatog. method was adapted for the assay of pantothenic acid in infant milk formulas. Sample prepn. consisted of **deproteination** with acetic acid and sodium acetate solns., followed by centrifugation and **filtration**. The chromatog. system included a C-18 column and a mobile phase consisting of a sodium phosphate buffer and **acetonitrile** (97:3, vol/vol). The column effluent was monitored by UV detection at 197 nm. The system was linear from 50 to 800 ng. The recoveries of pantothenic acid from augmented samples ranged from 89 to 98%, and the coeffs. of variation ranged from 1.17 to 3.20%. The results obtained with the HPLC and a microbiol. method were highly correlated for starting infant formula, followup infant formula, and formula for infants of low birth wt. from four different manufacturers. All formulas analyzed contained pantothenic acid at concns. higher than

those declared on their nutritional labels and were in compliance with international recommendations.

L6 ANSWER 32 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 113:90812 CA
OREF 113:15087a,15090a
TI Measurement of verapamil in human plasma by reversed-phase high-performance liquid chromatography using a short octyl column
AU Rustum, Abu M.
CS Dep. Environ. Fate Metab., Hazleton Lab. America, Inc., Madison, WI, 53707, USA
SO Journal of Chromatography, Biomedical Applications (**1990**), 528(2), 480-6
AB An HPLC method for the detn. of the Ca blocker verapamil in human blood plasma is described. The samples were **deproteinated** with **acetonitrile**/ZnSO4/MgSO4, centrifuged, and **filtered** prior to the anal. on a reversed-phase C-8 column. The anal. used a mobile phase of **acetonitrile**-phosphate buffer (60:40) adjusted to pH 7.1. The UV-detector was set at 220 nm. The calibration was linear in the range of 20-500 ng/mL, the accuracy was 95.0-98.3%, and the coeffs. of variation were 2.4-5.1%. No interference from plasma components and common drugs was found.

L6 ANSWER 33 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 111:70204 CA
OREF 111:11655a,11658a
TI A high-performance liquid chromatographic method for the measurement of deferoxamine in body fluids
AU Tesoro, Angelo; Leeder, J. Steven; Bentur, Yedidia; Klein, Julia; Freedman, Melvin; Koren, Gideon
CS Res. Inst., Hosp. Sick Child., Toronto, ON, M5G 1X8, Can.
SO Therapeutic Drug Monitoring (**1989**), 11(4), 463-70
AB A high-performance liq. chromatog. method for the anal. of deferoxamine (DFO) in 100 μL of serum or plasma is described. The procedure involves the addn. of the internal std. ciprofloxacin to the sample, followed by **ultrafiltration** to **remove protein**. The **ultrafiltrate** is then directly injected into the chromatog. system. Sepn. is achieved using a reverse-phase μBondapak C18 column and a ternary solvent system (sodium phosphate:**acetonitrile**:methanol) running at 2.0 mL/min. Assay time is 10 min, and chromatograms show no interference from coadministered drugs during this period of time. Coeffs. of variation were found to be less than 5%, and anal. recovery of DFO was 85%. Validation expts. in an exptl. dog model and in patients with iron overload demonstrate that the method is appropriate for studying the pharmacokinetics of DFO in thalassemic patients receiving drug for the treatment of chronic iron overload.

L6 ANSWER 38 OF 41 CA COPYRIGHT 2009 ACS on STN
AN 107:112183 CA
OREF 107:18127a,18130a
TI Deproteinizing methods evaluated for determination of uric acid in serum by reversed-phase liquid chromatography with ultraviolet detection
AU Sakuma, Ryozo; Nishina, Toshihiro; Kitamura, Motoshi

CS Dep. Clin. Chem., Toranomon Hosp., Tokyo, 105, Japan
SO Clinical Chemistry (Washington, DC, United States) (**1987**), 33(8), 1427-30
AB Six deproteinizing methods were evaluated for detn. of uric acid in serum by high-performance liq. chromatog. with UV detection: those involving zinc hydroxide, sodium tungstate, trichloroacetic acid, perchloric acid, **acetonitrile** and centrifugal **ultrafiltration** (with Amicon MPS-1 devices). A Toyosoda ODS-120A reversed-phase column was used. The mobile phase was sodium phosphate buffer (40 mmol/L, pH 2.2) contg. 20 mL of methanol per L. Absorbance of the eluate was monitored at 284 nm. The pptn. method with perchloric acid gave high recoveries of uric acid and good precision, and results agreed with those by the uricase-catalase method of N. Kageyama (1971).

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